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TECHNICAL REPORT NO. 1

TP-2, MSC-6621

SUPERPLASTIC DEFORMATION OF ZnS NEAR ITS

TRANSFORMATION TEMPERATURE (1020°C)

by

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Prepared for Publication

in the

Journal of the American Ceramic Society

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February 1, 1989

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## REPORT DOCUMENTATION PAGE

1a. REPORT SECURITY CLASSIFICATION		1b. RESTRICTIVE MARKINGS	
2a. SECURITY CLASSIFICATION AUTHORITY		3. DISTRIBUTION / AVAILABILITY OF REPORT	
2b. DECLASSIFICATION / DOWNGRADING SCHEDULE		Approved for public release, distribution unlimited.	
4. PERFORMING ORGANIZATION REPORT NUMBER(S) No: 1		5. MONITORING ORGANIZATION REPORT NUMBER(S) N00014-87-K-0527	
6a. NAME OF PERFORMING ORGANIZATION Cornell University	6b. OFFICE SYMBOL (If applicable)	7a. NAME OF MONITORING ORGANIZATION Office of Naval Research Dr. R. W. Schwartz	
6c. ADDRESS (City, State, and ZIP Code) Department of Materials Science & Eng'g. Ithaca, NY 14853-1501		7b. ADDRESS (City, State, and ZIP Code) Code 38504 NWC, China Lake, CA 93555	
8a. NAME OF FUNDING / SPONSORING ORGANIZATION	8b. OFFICE SYMBOL (If applicable)	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER	
8c. ADDRESS (City, State, and ZIP Code)		10. SOURCE OF FUNDING NUMBERS	
		PROGRAM ELEMENT NO.	PROJECT NO.
		TASK NO.	WORK UNIT NO.
11. TITLE (Include Security Classification) Superplastic Deformation of ZnS Near Its Transformation Temperature (1020°C)			
12. PERSONAL AUTHOR(S) Liang A. Xue and Rishi Raj			
13a. TYPE OF REPORT Technical	13b. TIME COVERED FROM TO	14. DATE OF REPORT (Year, Month, Day) 1, February 1989	15. PAGE COUNT 27
16. SUPPLEMENTARY NOTATION To be published in Journal of the American Ceramic Society.			
17. COSATI CODES		18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)	
FIELD	GROUP	SUB-GROUP	
		ZINC Sulfide	
19. ABSTRACT (Continue on reverse if necessary and identify by block number) Polycrystalline <del>ZnS</del> was deformed in compression to large strains (up to 100%), near its transformation temperature (1020°C), at strain rates that ranged from $10^{-5}$ s <sup>-1</sup> to $3 \times 10^{-3}$ s <sup>-1</sup> . The flow stress showed a minimum near 1020°C. The strain rate sensitivity and the grain size dependence of the flow stress suggest a superplastic mechanism of deformation. There was some evidence of dynamic recrystallization and stress induced <del>(fcc)</del> <sup>(hcp)</sup> phase transformation. The polycrystals had a tendency to form intergranular cavities at the higher temperatures and at faster strain rates. (mgm)			
20. DISTRIBUTION / AVAILABILITY OF ABSTRACT <input checked="" type="checkbox"/> UNCLASSIFIED/UNLIMITED <input type="checkbox"/> SAME AS RPT <input type="checkbox"/> DTIC USERS		21. ABSTRACT SECURITY CLASSIFICATION	
22a. NAME OF RESPONSIBLE INDIVIDUAL Rishi Raj		22b. TELEPHONE (Include Area Code) (607) 255-4040	
		22c. OFFICE SYMBOL	

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TEMPERATURE (1020°C)

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June 1988

Materials Science Report Number: 6621

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ABSTRACT

Polycrystalline ZnS was deformed in compression to large strains (up to 100%), near its  $\alpha \rightleftharpoons \beta$  transformation temperature (1020°C), at strain rates that ranged from  $10^{-5} \text{ s}^{-1}$  to  $3 \times 10^{-3} \text{ s}^{-1}$ . The flow stress showed a minimum near 1020°C. The strain rate sensitivity and the grain size dependence of the flow stress suggest a superplastic mechanism of deformation. There was some evidence of dynamic recrystallization and stress induced  $\beta$  (fcc) to  $\alpha$  (hcp) phase transformation. The polycrystals had a tendency to form intergranular cavities at the higher temperatures and at faster strain rates.

### INTRODUCTION

Deformation of crystalline materials near a phase transition temperature is interesting because of the possibility of unusual interactions between deformation and the change in microstructure. In magnesium-aluminate spinel, for example, the ductility of the material appears to be significantly enhanced just below the phase transition temperature (1); a similar effect has been observed near the  $\alpha + \beta$  phase change in titanium alloys (2). In both cases, dynamic recrystallization, which occurs more easily near the phase transition, appears to enhance ductility.

Zinc sulphide undergoes a phase transition from a face-centered-cubic to a hexagonal-close-packed structure at 1020°C. The fcc phase is called the  $\beta$  or the sphalerite phase and the hcp the  $\alpha$  or the wurtzite phase. In the present experiments hot-pressed specimens were deformed under uniaxial compression at true strain rates that ranged from  $10^{-5} \text{ s}^{-1}$  to  $3 \times 10^{-3} \text{ s}^{-1}$  at temperatures from 900°C to 1150°C. The flow stress was measured and the microstructure was characterized.

In the results reported below we show that the flow stress in ZnS changed with temperature in a way that led to a minimum in flow stress near the phase transition temperature. At lower temperatures the form of the flow curves resembled those obtained when deformation induces dynamic recrystallization in the polycrystal (3). Evidence was obtained for deformation-enhanced kinetics of the phase transformation from the cubic to the hexagonal phase.

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The strain rate sensitivity and the grain size dependence of the flow stress in ZnS satisfy the criteria for a superplastic deformation mechanism (4,5,6).

### EXPERIMENTAL

Hot pressed material was obtained from Eastman Kodak Co. The grain size was equiaxed and uniform, the average value being 1.9  $\mu\text{m}$ . Specimens for the deformation experiments were cut as parallelepipeds of approximate size 7 mm x 7 mm x 9 mm, or 8 mm x 8 mm x 12 mm. The tests were carried out in argon at one atmosphere pressure. The furnace elements were made from tungsten and the push rods used to deform the specimens were made from graphite.

The mechanical testing was done in uniaxial compression, at constant displacement rates. The displacement rates used were from 0.01 mm/min up to 2 mm/min. These corresponded to initial strain rates,  $|\dot{\epsilon}_z^0|$ , of  $10^{-5} \text{ s}^{-1}$  to  $10^{-3} \text{ s}^{-1}$  respectively.

Since the specimens were deformed to strains of up to 100%, the strain rates were corrected for the change in the length of the specimen, and the axial stress for the change in the cross section of the specimens. The following equations were used to calculate the true strain,  $\epsilon_z$ , strain rate,  $\dot{\epsilon}_z$ , and the true stress,  $\sigma_z$ :

$$\epsilon_z = \ln \frac{l}{l_0} \quad [1a]$$

$$\dot{\epsilon}_z = \frac{\dot{l}}{l} \quad [1b]$$

$$\sigma_z = \frac{P}{A_0} \cdot \frac{l}{l_0} \quad [1c]$$

where  $l$  is the time dependent height of the specimen,  $l_0$  is the initial height, and  $A_0$  is the initial cross sectional area. The crosshead displacement rate is  $\dot{l}$ , and the time dependent load measured during the experiment was  $P$ . The sign convention we use is that  $P$ ,  $\sigma_z$ , and  $\epsilon_z$  are negative if the loads and the strains are compressive, as is the case here.

## RESULTS

### Stress-Strain Curves

The change in the flow stress of ZnS with strain was obtained at several different temperatures and strain rates. Two sets of these results are given. Figure 1 shows the stress strain curves at 975°C, which is significantly below the transition temperature. The results at 1050°C, which is above the transition temperature, are given in Fig. 2. The deformability of the material at 1050°C is shown by a comparison between a deformed and undeformed specimen in Fig. 3; the total time required to deform the specimen was 4 min.

The results in Fig. 1 are well behaved, that is, the flow stress increases with the strain rate. The unusual characteristic of these curves is a maximum in the flow stress; this is reminiscent of studies on dynamic recrystallization of metals (7,8) where the maximum in the flow stress has been associated with the onset of the nucleation and growth of new grains. Deformation was also accompanied by extensive

twinning as shown by the micrograph in Fig. 4 from the specimen that was deformed at 975°C to a strain of -0.57 at a strain rate of  $9.4 \times 10^{-5} \text{ s}^{-1}$ .

The stress-strain behavior at 1050°C, shown in Fig. 2, differs markedly from 975°C. At small strain,  $\epsilon_z = -0.05$ , the flow stress increases systematically with strain rate. However, at larger strains the flow stress becomes irregular; for example at  $\epsilon_z = -0.60$  the flow stress does not correlate with the strain rate. A possible explanation for this erratic behavior is that the microstructure is unstable, and its evolution unpredictable, when the ZnS is deformed near its phase transition temperature.

The effects of deformation on the phase transformation from the fcc to the hcp phase at 1050°C, under different conditions of deformation, are described in Table I (the details of the x-ray technique used to measure the amount of the hcp phase are given later). Three cases are discussed, all having experienced approximately the same amount of strain, but each having been deformed at different strain rates, and therefore, for different periods of time. If deformation did not influence phase transformation then the volume fraction  $\alpha$  (hcp) phase would have been correlated only to the duration of the experiment. The data show, however, that the % hcp phase is correlated not to time, but to the stress (or the strain rate). We infer that high temperature, in situ deformation can influence the kinetics of phase transformation in ZnS.

The above result is reminiscent of another piece of work where ZnS was deformed at room temperature, and these specimens were annealed



at high temperature to study the effect of pre-strain on the rate of phase transformation. The pre-strain was found to lower the temperature for the onset of transformation from the fcc to the hcp phase (9).

The observation that deformed specimens of ZnS show extensive twinning in the fcc phase (Fig. 4) is further evidence of the influence of stress and strain on the phase change from the fcc to the hcp phase (the twin plane changes the local stacking from ABCABC to ABCABABC stacking where ABCABC stacking corresponds to the fcc phase and ABABAB to the hcp phase).

#### Strain Rate Sensitivity

The strain rate sensitivity measures the change in the flow stress when the material is deformed at different strain rates. Formally, it is defined by the following equation:

$$|\sigma_z| = A |\dot{\epsilon}_z|^m \quad [2]$$

where  $|\sigma_z|$  and  $|\dot{\epsilon}_z|$  are the magnitudes of the uniaxial stress and the uniaxial strain rate.  $A$  is a constant (it depends on the temperature and the microstructure, but not on the strain rate). The strain rate sensitivity index is  $m$ .

The parameter  $m$  is obtained experimentally by measuring the slope of a log-log plot of  $\sigma_z$  versus  $\dot{\epsilon}_z$ . Since the flow stress varies not only with strain rate but also with strain, the data must be gathered at a given value of the strain. The results for ZnS at different temperatures, and at  $\epsilon_z = -0.1$ , are given in Fig. 5. The slope leads

to  $m = 0.4$  at  $970^{\circ}\text{C}$ – $1100^{\circ}\text{C}$ , and  $m = 0.6$  at  $900^{\circ}\text{C}$  and  $950^{\circ}\text{C}$ . Even though the change in slope between these two temperature regimes is unmistakable the magnitude of the change is not significant enough to invoke different mechanisms of deformation.

A high value of the strain rate sensitivity provides resistance to strain localization, and, therefore, is a fundamental criterion for superplastic deformation. The highest possible value of  $m$  is 1, which describes Newtonian viscous flow, where the material is potentially infinitely ductile, like glass. Formal analysis (10) shows that reasonable superplastic ductility is possible even when  $0.4 < m < 1.0$ ; this has been verified in the deformation of a wide range of materials (11). The data in Fig. 5, therefore, meets at least one of the criteria for superplastic deformation in ZnS.

Unlike glass, crystalline materials have grain boundaries. In high temperature deformation, cavities may nucleate at these boundaries, which can limit the ductility of the material even if the strain rate sensitivity is within the bounds of superplastic flow as described above. In ZnS we found evidence of cavitation at high temperatures and fast strain rates. A micrograph of cavities from a specimen that was deformed at  $1150^{\circ}\text{C}$  at  $\dot{\epsilon}_z^0 = -3.7 \times 10^{-4} \text{ s}^{-1}$ , to a strain of  $\epsilon_z = -0.15$ , is shown in Fig. 6. Cavitation can be avoided by keeping the temperature below  $1050^{\circ}\text{C}$  and the strain rate below  $\dot{\epsilon}_z = -10^{-4} \text{ s}^{-1}$ . At higher temperatures, the increase in the flow stress (see Fig. 8) is probably the cause of cavitation.

### Deformation Mechanism

The important mechanisms of high temperature deformation in polycrystals are (i) the dislocation mechanism and (ii) the diffusional mechanism. They can be distinguished, experimentally, by studying the strain rate sensitivity ( $m$ ) and the grain size ( $d$ ) dependence of the flow stress. The dislocation mechanism usually leads to  $m < 0.25$ , and a flow stress that is independent of the grain size. In the diffusional mechanism,  $m = 1$ , and the flow stress varies as  $d^n$ , where  $n=2$  or  $3$ , depending on whether lattice diffusion ( $n = 2$ ) or grain boundary diffusion ( $n = 3$ ) is dominant.

Superplastic metals and alloys often behave as if both dislocation as well as diffusional mechanisms are operating simultaneously. One explanation invokes the idea that polydispersity of the grain size can cause mixed mode deformation in the polycrystal, where the large grains deform by dislocations while the smaller grains deform by diffusion (6). The model has also been applied to dynamic recrystallization where new grains are created as a result of dislocation deformation (5). The interesting feature of the mixed mode model is that it leads to a flow stress that is sensitive to the grain size (as required by the diffusional mechanism), but a strain-rate sensitivity that is less than 1.0, as a result of the dislocation mechanism. It is possible that these ideas are also applicable to large deformations in ZnS. We have already described that  $m < 1.0$  for ZnS. Below we provide evidence that the flow stress of the ZnS increases with the grain size.

The change in grain size during deformation is described by Fig. 7. The initial grain size was 1.9  $\mu\text{m}$ ; it increases to approximately 3.2  $\mu\text{m}$  at 950°C, but grows nearly eight fold at 1150°C. The data in Fig. 8 shows the change in the flow stress with temperature. The flow stress would have been expected to decrease exponentially with temperature if the deformation mechanism were grain size independent. In fact the flow stress reaches a minimum near 1050°C and then increases with temperature. This increase coincides with the rapid increase in grain size at temperatures above 1050°C, seen in Fig. 7, suggesting that flow stress was increasing with the grain size.

#### Dynamic Phase Transformation

The volume fraction of the  $\alpha$  (hcp) phase was measured by x-ray diffraction, using the relative intensities of the reflections from the (100) and (002) planes of hcp ( $\alpha$ ) structure, and the (111) plane of the fcc ( $\beta$ ) structure. The Bragg angle of the (002) plane of the  $\alpha$  phase ( $2\theta = 28.50^\circ$ ) lies near the angle for the (111) plane of the  $\beta$  phase ( $2\theta = 28.56^\circ$ ). We estimated the volume fraction of the  $\alpha$  phase using the same procedure that was employed by Bansagi et al. (12).

Since the Bragg diffraction from the  $\alpha(002)$  plane the  $\beta(111)$  plane overlap at  $2\theta = 28.53^\circ$ , the relative mole fraction of the  $\alpha$  phase is described by following parameter:

$$\gamma = \frac{I_{28.53}^{\{\alpha(002) + \beta(111)\}}}{I_{28.53}^{\{\alpha(002) + \beta(111)\}} + I_{26.91}^{\alpha(100)}} \quad [3]$$

Equation [3] has the following limits: When  $\beta = 0$ , then  $\gamma = \gamma_0$  where:

$$\gamma_0 = \frac{I_{28.53}^{\alpha(002)}}{I_{28.53}^{\alpha(002)} + I_{26.91}^{\alpha(100)}} \quad [4]$$

On the other hand when  $\beta = 1$ , that is  $\alpha = 0$ , then  $\gamma = 1$ . Here  $\alpha$  and  $\beta$  are being used to describe the mole fraction of each phase; hence  $\alpha + \beta = 1$ . Further analysis rests on the assumption (12) that an increase in  $\beta$  from 0 to 1 produces a linear increase in  $\gamma$  from  $\gamma_0$  to 1 which leads to the following equation:

$$\beta = \frac{\gamma - \gamma_0}{1 - \gamma_0} \quad [5]$$

or that

$$\alpha = 1 - \beta = \left( \frac{1}{1 - \gamma_0} \right) (1 - \gamma) \quad [6]$$

The value for  $\gamma_0$  may be obtained from Eq. [4] but substituting the intensity values reported in ASTM diffraction file Index Nos. 5-0566 and 36-1450, which gives that  $\gamma_0 = 0.4565$ . Making this substitution in Eq. [6] we obtain that:

$$\alpha = 1.84 (1 - \gamma) \quad [7]$$

The results for phase transformation from the fcc ( $\beta$ ) phase to the hcp ( $\alpha$ ) phase in the specimens deformed at different temperatures for different lengths of time, are reported in Fig. 9. It is

interesting that the acceleration in the phase transformation, the minimum in the flow stress, and the onset of rapid grain growth, all occur at approximately 1050°C. The minimum in the flow stress can be explained in terms of the rapid grain growth, but that correlation is a little ambiguous since an increase in the volume fraction of the hcp may also have caused the flow stress minimum if the hcp phase is more difficult to deform than the fcc phase.

Transformation induced superplasticity in ceramics is a subject that has been sporadically reported in the literature since the early sixties starting with the work of Chaklader and coworkers. In one type of experiments the ceramic is cycled across a phase transition temperature under an applied load. Every cycle produces incremental strain which can accumulate to large elongations (for example see Ref. 13). In the second type of experiments, first introduced by Morgan and Scala (14) reactions during hot-pressing have been shown to produce unusually fast rates of densification. A phenomenological interpretation of our results is similar since we too obtain the lowest value of the flow stress just below the transformation temperature. Our interpretation, however, is more straightforward. It is that the rapid growth in the grain size above the transition is the reason for the minimum in the flow stress.

In our view the value of dynamic phase transformation lies in alleviating the stress concentration at grain boundaries through a local change in phase--since a change in phase can accommodate and relax a local shear and/or hydrostatic stress. Thus phase change can enhance ductility. If the phase change also reduces the grain size

then the flow stress for diffusional creep may also be reduced. Metallurgical evidence for this mechanism was presented in similar experiments with a titanium alloy (2).

#### CONCLUSIONS

1. Zinc sulphide deforms with characteristics of superplastic deformation at temperatures near the  $\beta \rightarrow \alpha$  phase transition.
2. At very high strain rates and high temperatures, the material is prone to intergranular cavitation.
3. There is some evidence that ZnS recrystallizes dynamically when deformed just below the phase transition temperature.
4. There is good evidence that the phase transformation from the fcc to the hcp phase is accelerated by in situ deformation.

#### ACKNOWLEDGEMENTS

This research was supported by the Office of Naval Research. Support was also received from the Materials Science Center through the use of the facilities of the Materials Science Center.

FIGURE CAPTIONS

- Fig. 1. Stress strain curves for ZnS at 975°C. The peak in the flow stress suggests the onset of dynamic recrystallization.
- Fig. 2. Stress strain curves for ZnS at 1050°C. The peak in the flow stress suggests the onset of dynamic recrystallization.
- Fig. 3. Undeformed specimen (A) and a specimen (B) after deformation for 4 min at 1050°C.
- Fig. 4. Micrograph of a specimen deformed at 975°C at an initial strain rate of  $9.4 \times 10^{-5} \text{ s}^{-1}$ . Note the extensive twinning.
- Fig. 5. Determination of the strain rate sensitivity index,  $m$ , by plotting  $\sigma_z$  vs  $\dot{\epsilon}_z$  at a constant strain, on a log-log scale.
- Fig. 6. At high strain rates and high temperatures intergranular cavities nucleate and grow in polycrystalline ZnS. This specimen was deformed at 1150°C at a strain rate of  $3.7 \times 10^{-4} \text{ s}^{-1}$ , to a strain of  $\epsilon_z = -0.15$ .
- Fig. 7. Grain growth in ZnS during deformation at different temperatures.
- Fig. 8. The change in flow stress with temperature at approximately constant strain-rate, and at a fixed value of the strain.
- Fig. 9. The extent of phase transformation from the fcc to the hcp phase in specimens deformed at different temperatures for different lengths of time. Note that the phase change accelerates at the same temperature where the flow stress exhibits a minimum (Fig. 8).



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Temp. (°C):	900	950	975	1000	1050	1100	1150
Time (min):	100	70	105	105	120	30	11
$-\epsilon_o$ (s <sup>-1</sup> ):	0.7	1.4	0.94	0.94	0.96	3.6	$3.6 \times 10^{-4}$
$-\epsilon_z$ :	0.33	0.56	0.57	0.59	0.80	0.66	0.15

Temp. (°C):	950	950	950	950	1050	1050	1100	1100	1150
Time (min):	70	130	265	440	4	120	5	30	11
$-\epsilon_o$ :	0.56	0.43	0.59	0.24	0.75	0.81	0.51	0.66	0.15
$-\epsilon_z$ :	1.40	0.56	0.17	0.11	28.0	0.96	14.0	3.6	$3.6 \times 10^{-4}$

Table I. Phase change from the fcc to the hcp phase during deformation at 1050°C. Note that the mole % hcp is related to the flow stress. If deformation were not a factor then mole % hcp would have correlated to time.

Initial Strain Rate $\dot{\epsilon}_z$ (s <sup>-1</sup> )	Flow Stress ( $\epsilon = 0.05$ ) $\sigma_z$ (MPa)	Deformation (min)	Deformation Strain	Mole % hcp Phase
$9.6 \times 10^{-5}$	23	120	-0.81	8.3
$5.6 \times 10^{-4}$	50	11	-0.4	10.5
$2.8 \times 10^{-3}$	123	4	-0.75	14.6

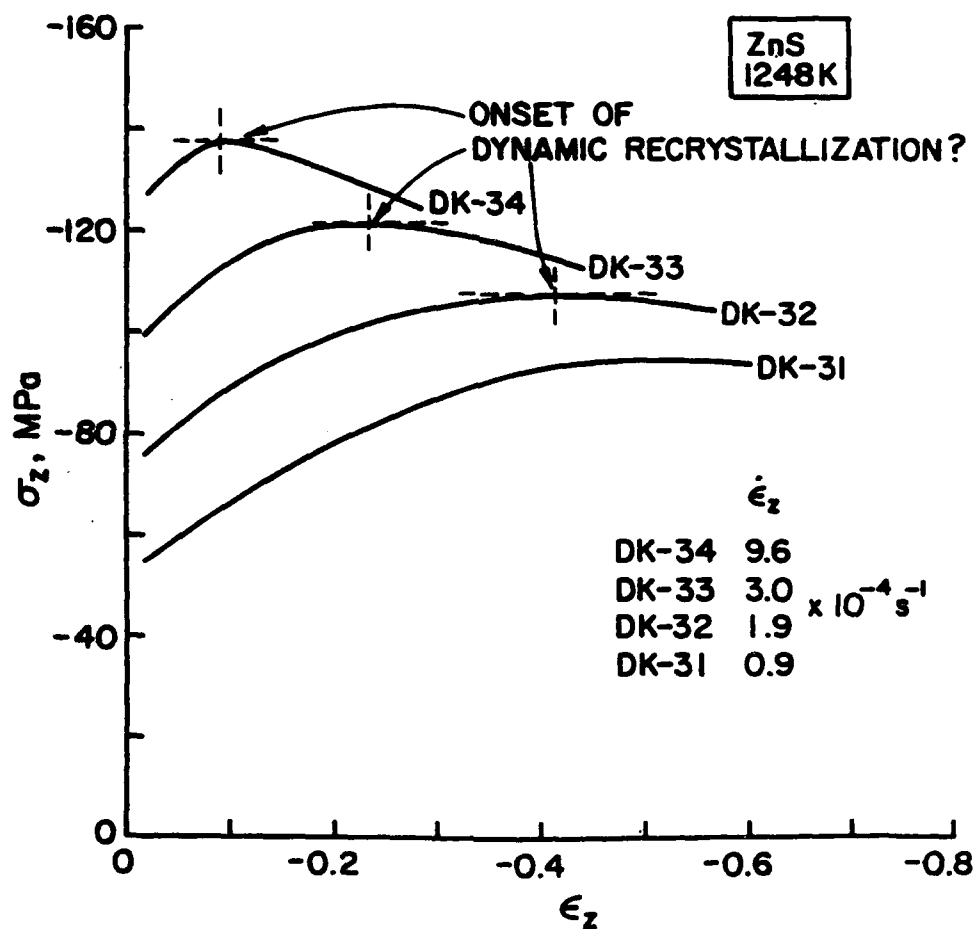


Fig. 1. Stress strain curves for ZnS at 975°C. The peak in the flow stress suggests the onset of dynamic recrystallization.

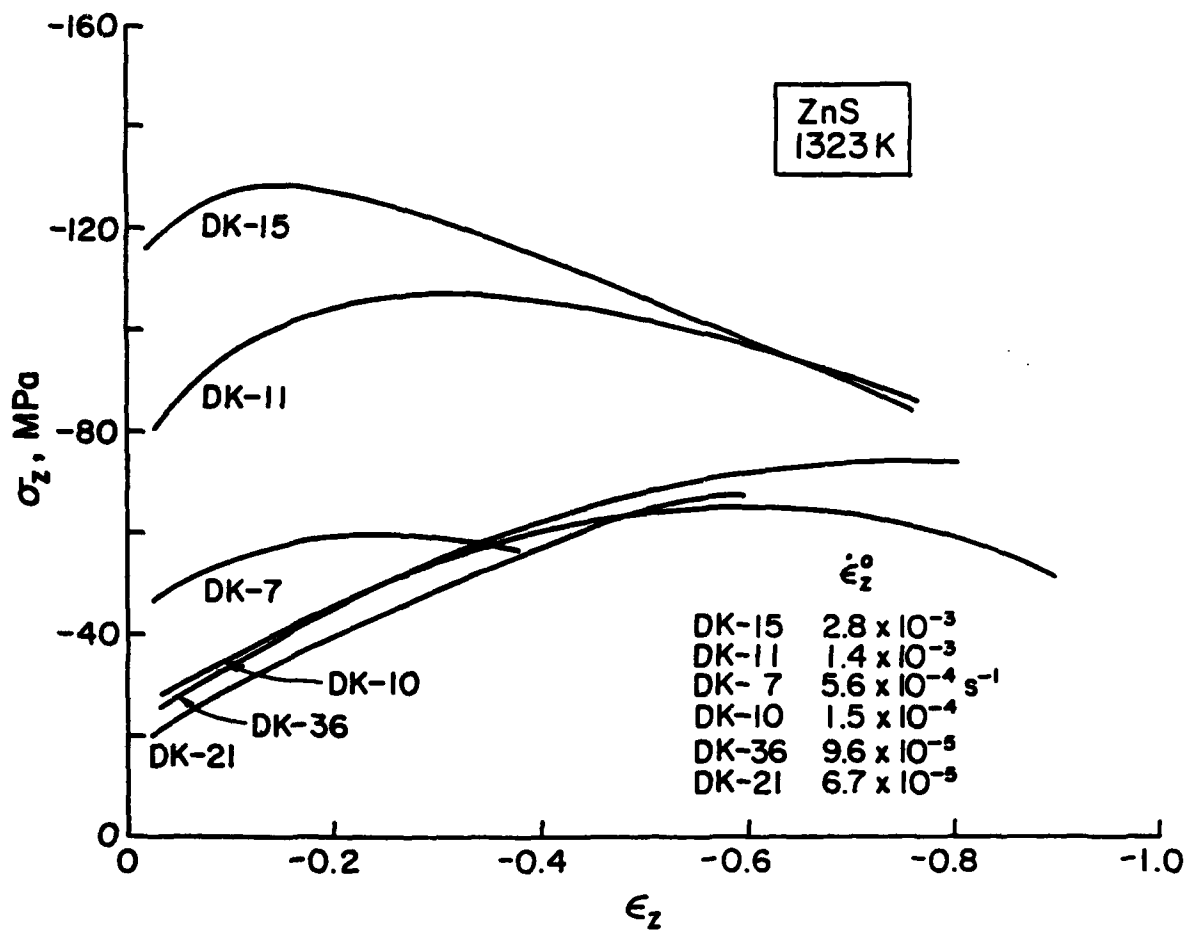


Fig. 2.

Stress strain curves for ZnS at 1050°C. The peak in the flow stress suggests the onset of dynamic recrystallization.

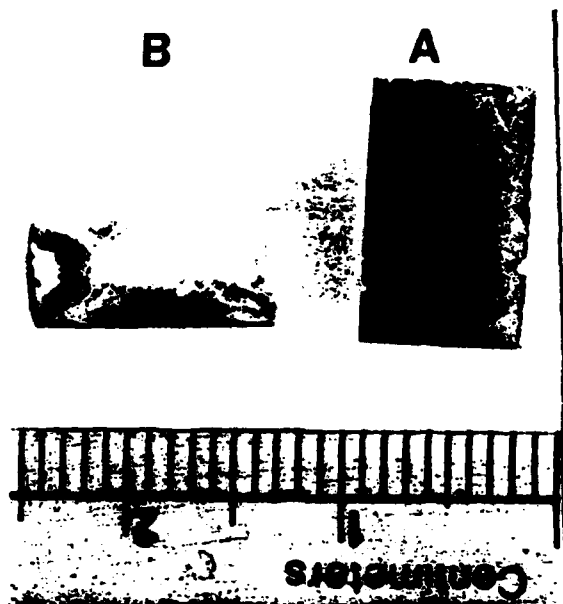


Fig. 3. Undeformed specimen (A) and a specimen (B) after deformation for 4 min at 1050°C.

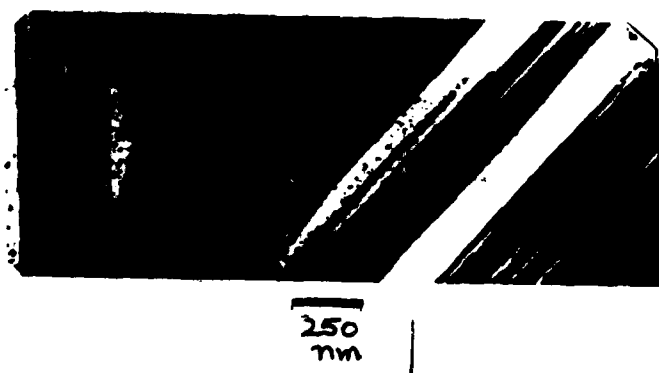


Fig. 4. Micrograph of a specimen deformed at 975°C at an initial strain rate of  $9.4 \times 10^{-5} \text{ s}^{-1}$ . Note the extensive twinning.



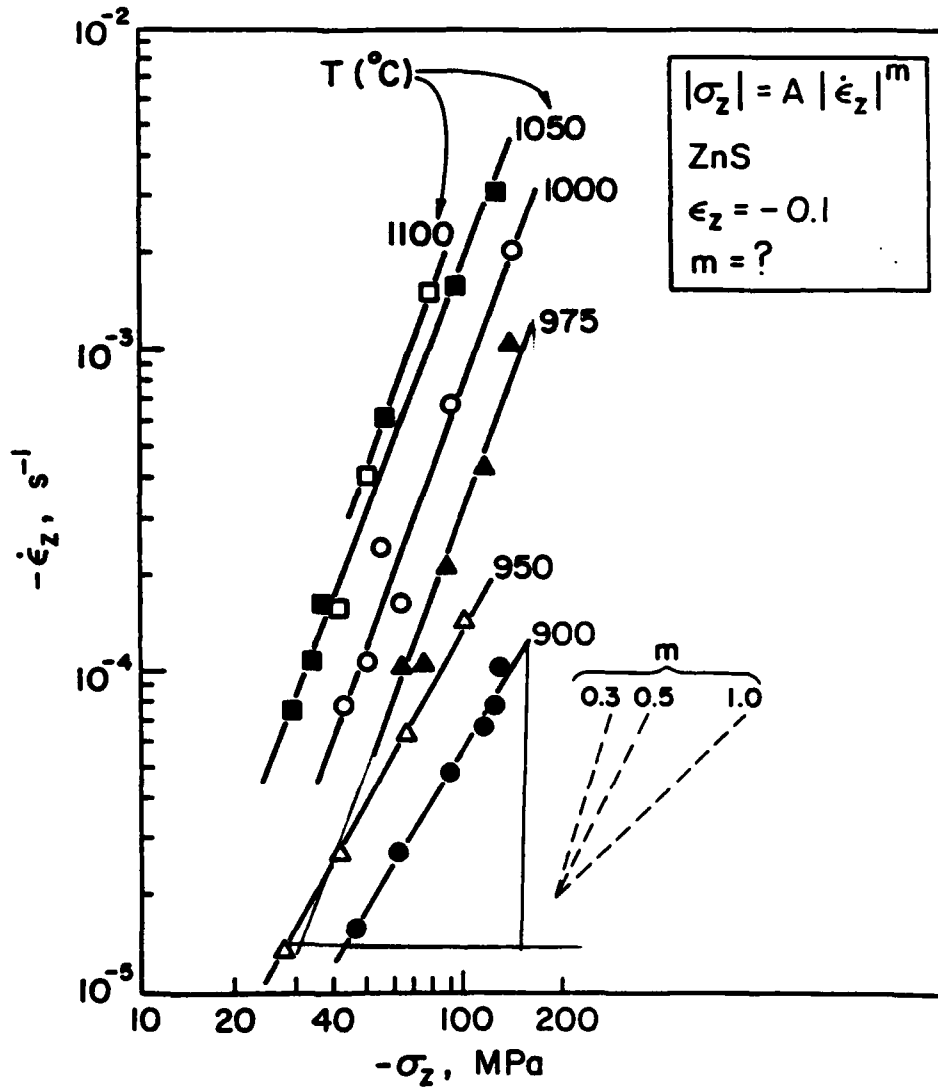
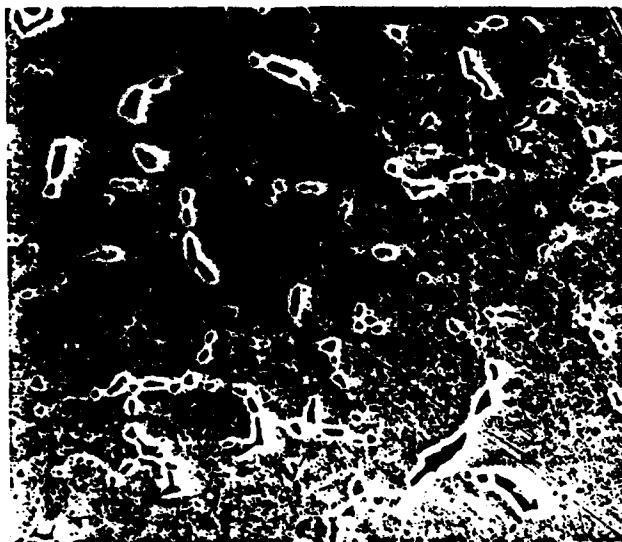


Fig. 5. Determination of the strain rate sensitivity index,  $m$ , by plotting  $\sigma_z$  vs  $\dot{\epsilon}_z$  at a constant strain, on a log-log scale.



**Fig. 6.** At high strain rates and high temperatures intergranular cavities nucleate and grow in polycrystalline ZnS. This specimen was deformed at 1150°C at a strain rate of  $3.7 \times 10^{-4} \text{ s}^{-1}$ , to a strain of  $\epsilon_z = -0.15$ .

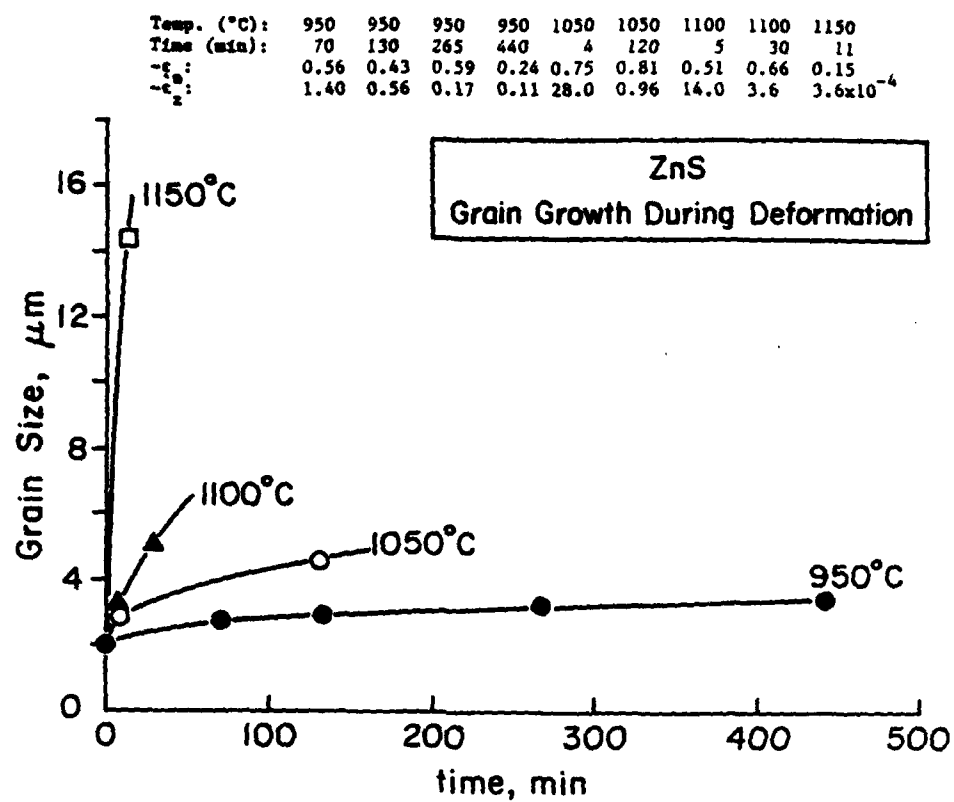


Fig. 7. Grain growth in ZnS during deformation at different temperatures.

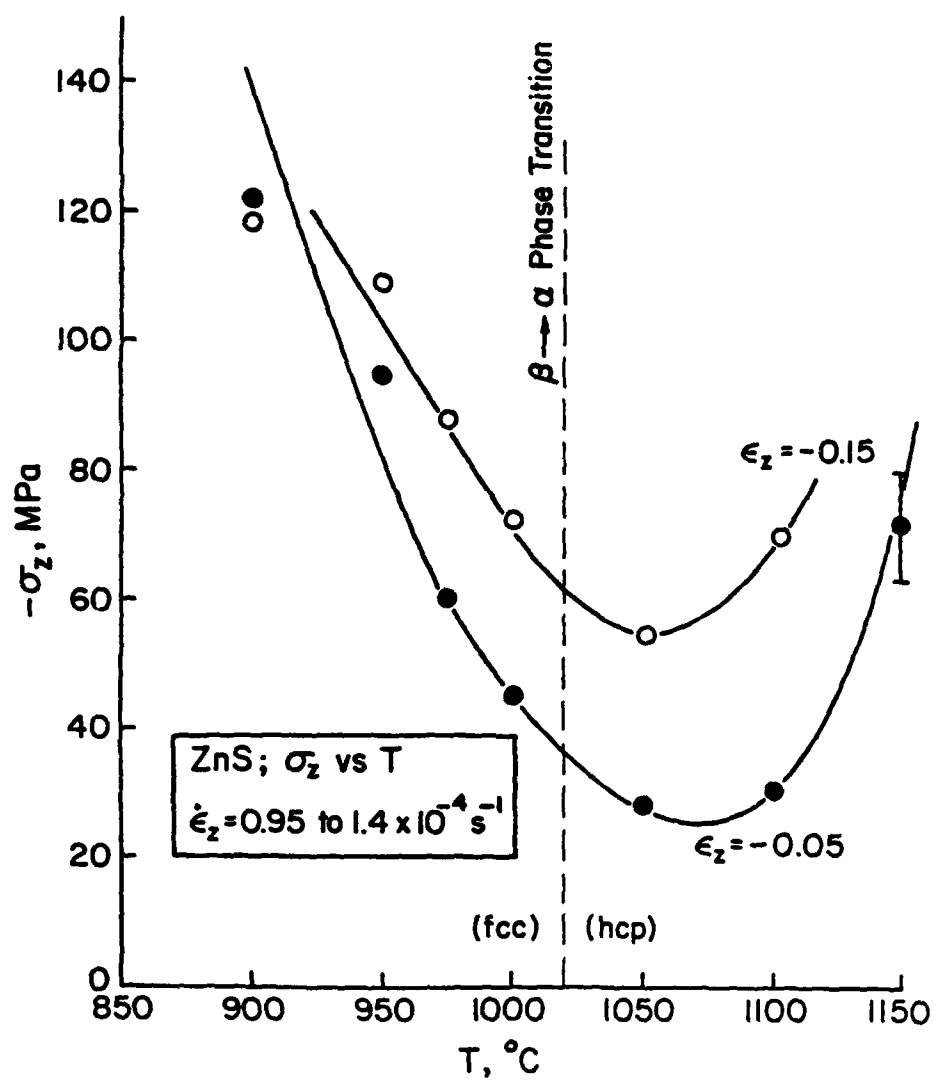


Fig. 8. The change in flow stress with temperature at approximately constant strain-rate, and at a fixed value of the strain.

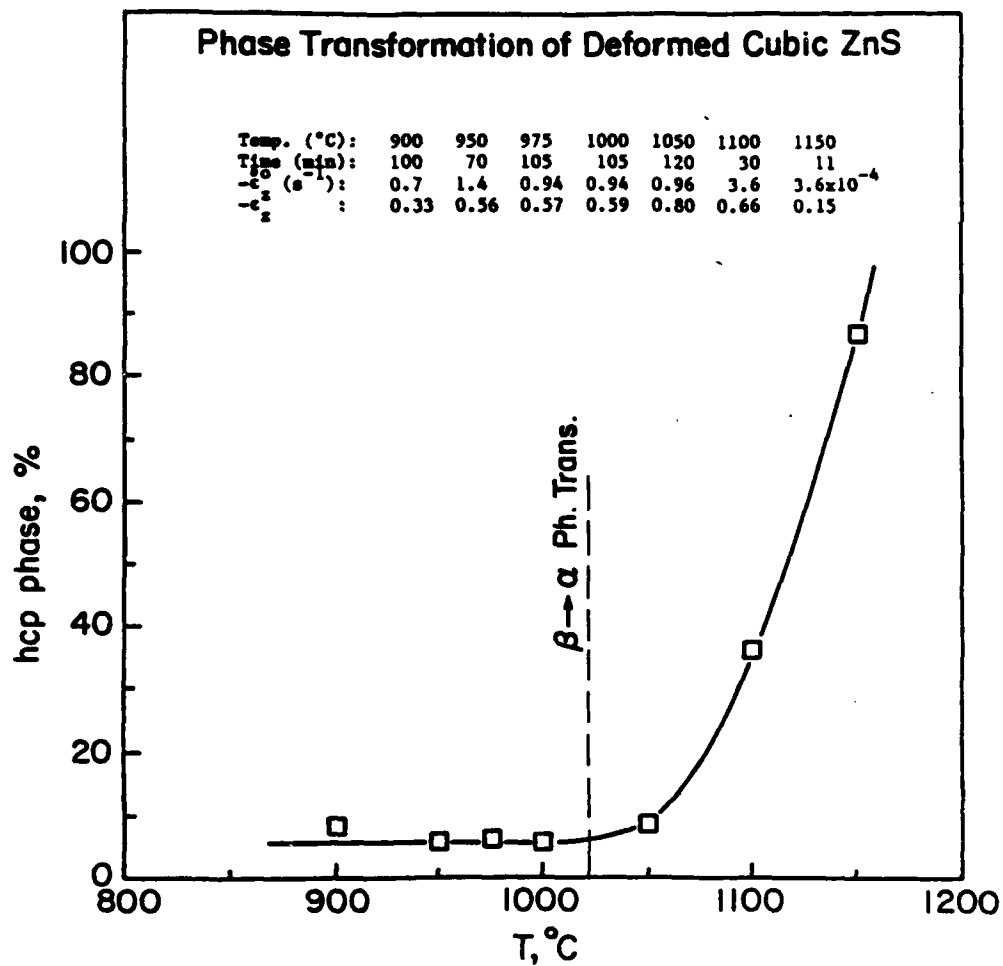


Fig. 9. The extent of phase transformation from the fcc to the hcp phase in specimens deformed at different temperatures for different lengths of time. Note that the phase change accelerates at the same temperature where the flow stress exhibits a minimum (Fig. 8).